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PYROMETRY OF THE PROCESSES OF SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS

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The developed method of luminance pyrometry used for temperature measurement in processes of self-propagating high-temperature synthesis, plasmathermal spraying of both moving and motionless media has been described. MIS photodiodes operating in direct detection mode or charge accumulation are sensitive elements of optoelectronic track of the device. Change of light signal intensity created by surface of reacting substances is recorded in two microareas. Research results of temperature, distribution speed of combustion front from time and quantitative content of titanium carbide in a batch as well as hardness of sintered samples of the Fe_2O_3, Al+TiC system are given.

Efficient complex methods of testing and recording thermal temperature-speed characteristics are still actual at physical researches of dynamics of development and mechanisms of forming products of self-propagating high-temperature synthesis (SHS). Acceleration of pace of development of range of products made on the basis of composite and intermetallic compounds requires growth of technological instrumentation of research laboratories and industrial productions. Application of high-quality diagnostic devices allows the researchers to transfer to the category of controlled reactions those ones which were uncontrolled or weakly controlled thermochemical reactions of SHS.

The self-propagating high-temperature synthesis discovered by academician A.G. Merzhanov is characterized by such unique feature as existence during some time the reaction of high-temperature solid-liquid medium permitting different types of influence (electrical, chemical, thermodynamic etc.) [1]. Besides this influence various alloying and inert additions allowing both changing synthesis mode and controlling structure and properties of the end product may be used in SHS-systems [2]. To study the interactions of these effects on thermalphysic processes of synthesis the recorders of dynamic optical fields – pyrometers can be applied. They are made on the basis of discrete and multiple-unit photodetectors functioning in direct detection mode or in charge storage mode. The main feature of such devices is contactlessness and low inertia of measurements in comparison with contact temperature-sensitive elements that promotes carrying out heat monitoring of fast high-energy processes with high accuracy. Investigations of temperature dynamics and processes of heat-and-mass transfer are complicated due to nonapplicability of contact methods of measuring temperature in dispersion weak packaged media in the range of 50...70 % of porosity that is typical for samples with packed density. In optical pyrometry of weakly luminous objects (in this case mixtures of powders or heterophase jet) measurements in the field of low temperatures are characterized by poor accuracy.

Measurement of luminance temperature and speed of product synthesis in combustion zone of high-energy mixtures is connected with a number of problems. Reactionary cell has a small size (100...500 mkm), its occurrence in sight of pyrometer is of stochastic nature. Synthesis should be carried out very often in the form of end item; in this case reactor wells can not be always made of optical-transparent material. Methods of optical pyrometry allow determining thermalphysic parameters on the surface of synthesized article [3] they are not capable of providing the researcher with information on structure of combustion in deep layers of the product without assuming additional measures. However, these disadvantages of pyrometric methods are not insurmountable ones and they may be easily overcome with sufficient part of probability in laboratory environment that allows obtaining precise information on the process of synthesis [3, 4].

The aim of the work was to develop the method and recording system for diagnostics of thermal parameters of high-energy mixtures on the basis of steel cinder with alloying additions as well as to obtain the dependences of such mixture combustion rate on various admixtures.

For recording the temperature of combustion front the fast rate and temperature meter was developed. This device allows measuring by time-of-flight method of integral and local rates, estimating integral temperature distribution in two points, measuring combustion front temperature with contact ratio correction, determining average speed of combustion wave propagation of SH-synthesis of disperse material mixture, as well as measuring temperature dynamics of high-energy mixture reaction.

Pyrometer shown in Fig. 1 is constructed on the basis of optical photo-diode camera which contains: optical system serving for adjustment of optical channels, telescopic adapter PZF-1 where the television objective, the tray for setting filter wheels (KS-18,KS-19, IKS-3 etc.) are installed. Two silicon photodiodes FD-256A are used as photodetectors. Apertures specifying photodetector sight to 100 mkm on object surface are imposed on them. Optical head contains also two converters «current-voltage», two logarithmic amplifiers of the first order linearizing output signal, power amplifiers coordinating output circuits of FD-camera with 50-ohm inputs of analog-digital converter made on the basis of DAB built-in PC (model: LA-n20-PCI, manufacturer: «Rudnev-Shilyaev»); system of direct current balancing of channels and circuit of compensation of photodiode dark current.

Block of analog-digital processing and synchronizing implementing functions of analog-digital conversion, calculation of contact ratio, signal normalizing, production of synchrosignals for internal nodes and

external devices, stores the obtained information and transfers it through the back-end interface (bus PCI) into controlling computer. Pyrometer temperature calibration was carried out by a standard technique using temperature lamp TRU-1100. Diagram of dependence of luminance and thermodynamic temperature of etalon on current (I) flowing through it and diagram of transformation of ADC gradation (N_{adc}) into temperature are given in Fig. 2.

Fig. 1. *Pyrometric complex*

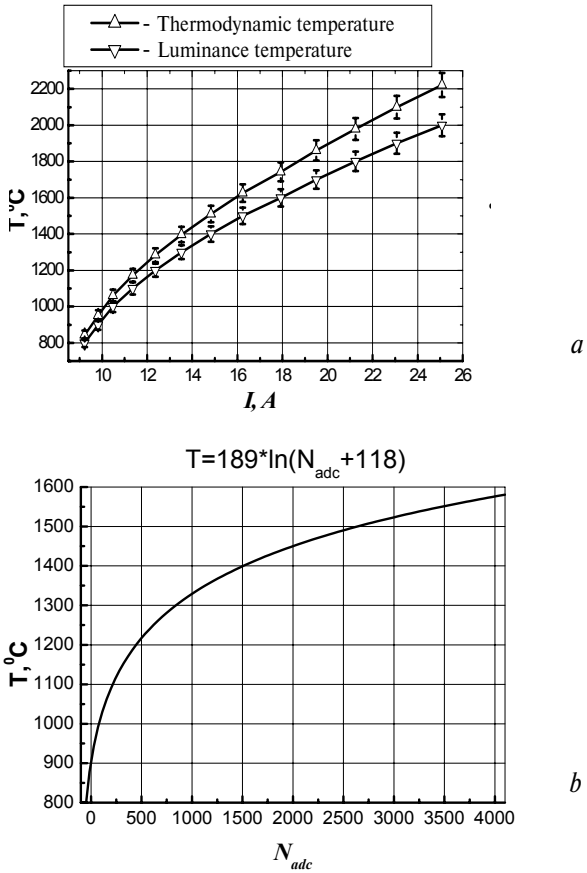

Fig. 2. *Luminance and thermodynamic temperatures of etalon lamp TRU-1100 (a), pyrometer gauge function (b)*

Current in a circuit of photodiode is proportional to voltage $U_{\phi\partial}$ on photodiode. According to [4, 5] voltage on photodiode in accumulation mode may be presented as:

$$\frac{U_{\phi\partial}}{U_0} = \left[1 - A\int_0^{t_н} P_\lambda(t)dt + \frac{A^2}{2}\left(\int_0^{t_н} P_\lambda(t)dt\right)^2\right], \qquad (1)$$

where $A = \dfrac{e \cdot S_{\phi\partial} \cdot \eta}{2h\nu C_0 U_0}$, e is the electron charge; $S_{\phi\partial}$ is the area of photodetector and η is its quantum yield; C_0 is the initial capacity of photodiode; U_0 is the voltage of reverse bias source; $P_\lambda(t)$ is the density of luminous flux at wave length λ, $t_н$ is the time of charge storage by photodiode.

Thresholds are selected in discharge linear region when quadratic term in (1) may be neglected. Let us express integral in the expression (1) as:

$$\int_0^{t_н} P_\lambda(t)dt = \frac{1}{A}\left(1 - \frac{U_{\phi\partial}}{U_0}\right).$$

To a first approximation let us consider flux $P_\lambda(t)$ to be constant during accumulation time $t_н$, then $P_\lambda(t)$ may be calculated as:

$$P_\lambda = \frac{f(U_{\phi\partial}, U_0)}{t_н}.$$

Defining the time of photodiode discharge from the original value U_0 to the specified value $U_{\phi\partial}$ that corresponds to current change in photodiode circuit from I_0 to $I_{\phi\partial}$ we can measure luminous flux density at fixed wave length. Current $I_{\phi\partial}$ is specified initially – fixed previously selected threshold, current I_0 is determined by photodiode output parameters by a number of attached channels and reversal spectrum U_0; respectively, the value $f(U_{\phi\partial}, U_0)=F$=const.

On the other side, flux is proportional to the area of radiating surface and depends on its temperature and radiative characteristics. Let us write down in Vin approximation:

$$\Phi \sim S_0 P_\lambda \sim S_0 \varepsilon_\lambda c_1 \lambda_i^{-5} \exp\left(-\frac{c_2}{\lambda_i \cdot T}\right),$$

where: S_0 is the area of the observed surface, T is the surface temperature, c_1, c_2 are the constants. The ratio of fluxes from one object at two different wave length does not depend on sighted area:

$$\frac{P_{\lambda 1}}{P_{\lambda 2}} = \frac{\varepsilon_{\lambda 1}}{\varepsilon_{\lambda 2}}\left(\frac{\lambda_1}{\lambda_2}\right)^{-5} \exp\left(\frac{c_2}{\lambda_2 T} - \frac{c_2}{\lambda_1 T}\right) = \frac{F_1 t_{н2}}{F_2 t_{н1}}.$$

Converting this expression we obtain the ratio for determining temperature:

$$T = \frac{C}{\ln\dfrac{t_{н2}}{t_{н1}} + B}, \qquad (2)$$

where

$$C = c_2\left(\frac{1}{\lambda_2} - \frac{1}{\lambda_1}\right), \quad B = \ln\frac{F_1}{F_2} \cdot \frac{\varepsilon_{\lambda 2}}{\varepsilon_{\lambda 1}}\left(\frac{\lambda_1}{\lambda_2}\right)^5.$$

Thus, it is seen from the expression (2) that temperature is inversely proportional to logarithm of ratio of charge storage times on photodiodes. Measuring specified times on different wave lengths the information on object temperature may be obtained.

Relative error of determining temperature by this method:

$$\frac{\Delta T}{T} = \frac{T}{C}\sqrt{\left(\frac{\Delta t_{н1}}{t_{н1}}\right)^2 + \left(\frac{\Delta t_{н2}}{t_{н2}}\right)^2 + (\Delta B)^2},$$

where

$$\Delta B = \sqrt{\left(\frac{1}{F_1}\Delta F_1\right)^2 + \left(\frac{1}{F_2}\Delta F_2\right)^2 + \left(\frac{1}{\varepsilon_{\lambda 1}}\Delta\varepsilon_{\lambda 1}\right)^2 + \left(\frac{1}{\varepsilon_{\lambda 2}}\Delta\varepsilon_{\lambda 2}\right)^2},$$

ΔF_i is the change of photodetector parameters caused by temperature instability, reference voltage fluctuations etc.; $\Delta\varepsilon_{\lambda i}$ is the error of determining radiation capacity of the observed surface (for example, owing to oxidation, contamination etc.)

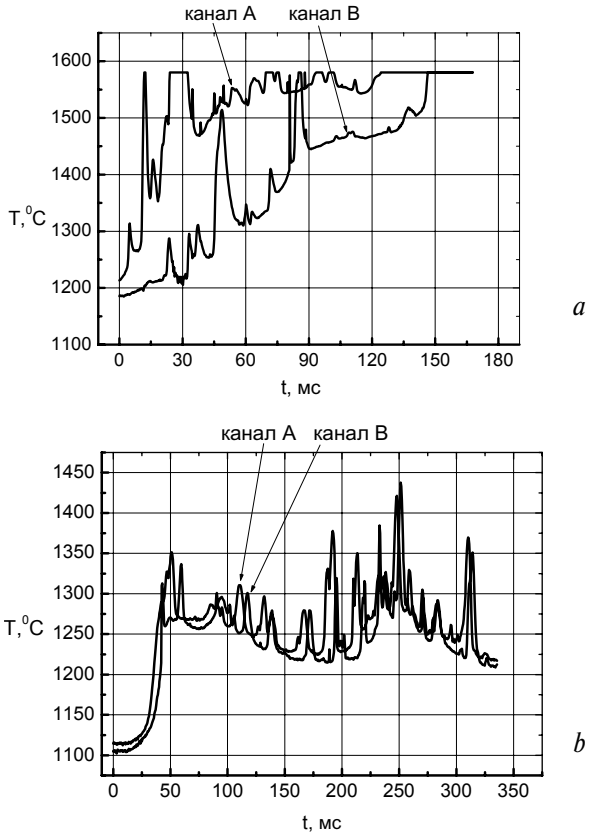

Fig. 3. *Typical diagrams of temperature change in combustion wave of SHS mixture Fe_2O_3, Al+TiC: a) 5 % TiC, b) 15 % TiC*

To carry out the experiment the original batch (mixture of Fe_2O_3, Al and TiC) was placed in steel equipment having rectangular hole for setting quartz glass holders in it through which luminance temperature of batch surface is recorded or the original batch is placed in quartz cylinder. The SHS process of tool steel was observed in the form of torch with spraying drops of melt; simultaneously data on thermal structure of synthesis process were recorded and digitized. Temperature profiles of combustion in two points arranged at a distance of 15 mm from each other vertically (channels A and B) are given in Fig. 3.

Ignition of SHS process is implemented by initiating heat pulse from a small amount of titanium boride being in upper part. Temperature dynamics and thermal field on batch surface are recorded by photodiode camera of pyrometer of by the camera of pyrometer-thermal imager. Optical camera and equipment with the batch are set at optical bench. Focusing on batch surface is carried out through pyrometer optical system. The required distance between the camera and equipment, diaphragm of camera optical system are determined. Program of recording in microprocessor unit is started, the required frequency of discretization is entered, light filter is arranged. Then titanium boride is initiated.

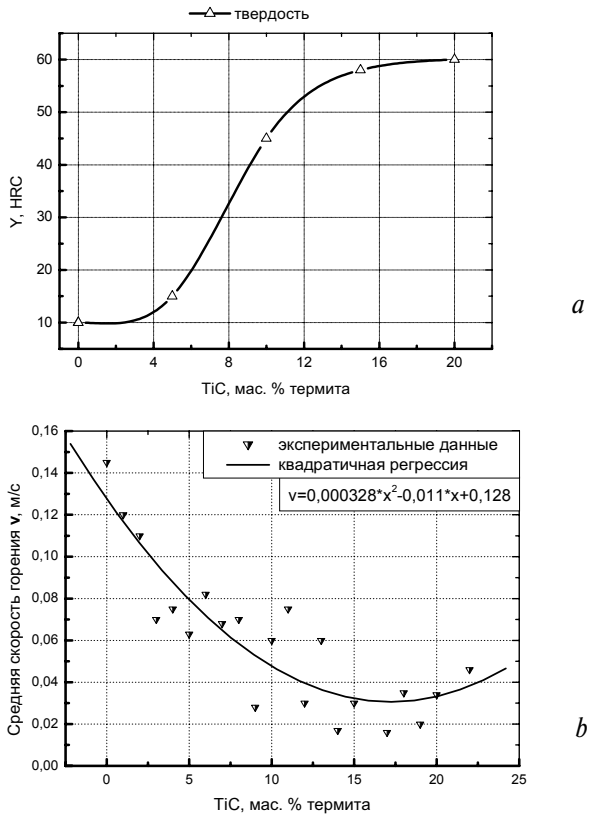

Fig. 4. *Hardness (a) and average rate of combustion (b) of the system Fe_2O_3, Al+TiC depending on mass fraction of titanium carbide*
Твердость – hardness; экспериментальные данные - experimental data; квадратичная регрессия – square regression

At the moment of combustion front passing through the first channel pyrometer sight (channel A) the program of digitization and recording of video signal from the process of SHS-synthesis are automatically started in RAM. Then microprocessor unit calculates contact ratio by video signal form, normalizes numerical data. After cooling-down the sintered specimen being in the equipment is placed into muffle furnace for recalibration of pyrometer indices. It is seen from the obtained thermograms that temperature in the process of interac-

tion has explicitly non-stationary character, growth periods are replaced by stabilization time and even decrease as the reaction cell in the process of synthesis undergoes a number of transformations. Exothermal regions replace the areas with endothermic decay.

The obtained samples of sintered material were tested for hardness. By the results of the experiments and the character of point arrangement the dependences (Fig. 4) of alloy hardness and rate of combustion front propagation on quantitative content of titanium carbide in the batch are plotted.

At increase of titanium carbide content in the batch the alloy hardness grows and combustion front rate decreases by nonlinear law and the character of dependences changes practically at one and the same value of titanium carbide mass in the batch (4...12 wt. % termite).

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CHANGE IN STABILITY OF SOLID SOLUTION AT RADIATION INFLUENCE

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Stability of solid solution at radiation influence has been investigated. Expressions for diffusion streams of binary alloy components, which specify the existence of temperature interval in which the phenomenon of ascending diffusion of elements is observed, were received. Vacancy characters of diffusion, configuration entropy, and potential energy of atomic bonds were considered at derivation. The ascending diffusion testifies to stability infringement of homogeneous solid solution – stratification. Influence of radiation is connected with increase in concentration of vacancies which changes the energy of atomic bonds and, simultaneously, accelerates diffusion processes. The condition of alloy stability with regard to stratification at radiating influence was obtained.

Radiation of metallic systems with charged particles or neutrons may result in some cases in significant changes of material structural-phase state. Radiation changes temperature of existence of one or another phase, results in change of stability of solid solutions (element segregation, phase dissolution etc.), causes occurrence of new exotic phases [1].

Accumulation of excess vacancies in the volume causing the occurrence of macroscopic elastic tensile stresses is typical for radiation influence. All principle experimentally observed laws of radiative swelling of fine metals were explained by bounded solubility of the excess vacancies in metal matrix [2–4]. From this point of view the solution of the problem of changing temperature of solid solution stability field at radiation is of interest. It is necessary to determine in what way the increased concentrations of vacancies may influence the layering of initially homogeneous solid solution.

1. Diffusive constituent of element streams

Let us consider binary alloy with concentrations of elements n_a and n_b. Let atoms of alloy components be transfered by vacancy mechanism. Diffusive constituents of atom streams are determined by the expressions

$$J_a = \frac{D_a}{n_0}[-n_v \cdot \mathrm{grad}(n_a) + n_a \cdot \mathrm{grad}(n_v)],$$

$$J_b = \frac{D_b}{n_0}[-n_v \cdot \mathrm{grad}(n_b) + n_b \cdot \mathrm{grad}(n_v)]. \qquad (1)$$

Here J_a, J_b are the diffusion flows of elements of alloy a and b respectively, D_a, D_b are the coefficients of element diffusion, n_v, n_0 are the concentrations of vacancies and sites of crystal lattice respectively. Equations (1) are obtained by standard method of three planes provided the independence of diffusion process for some elements of alloy.